

# CAPP

International Research Project on the Effects of Chemical  
Ageing of Polymers on Performance Properties

## CORRELATION OF CHEMICAL AND PHYSICAL TEST DATA FOR THE ENVIRONMENTAL AGEING OF TEFZEL (ETFE)

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## **SYNOPSIS**

Data from the many different tests conducted on Tefzel by MERL and TRI are reviewed herein with the objectives of identifying any trends or correlations between them. This report applies a similar approach to that in CAPP/M.10, which performed this task for Coflon. Some discussion on underlying mechanisms is included, and a rationale for the overall behaviour is suggested.

No evidence has been found to date that Tefzel degrades chemically when immersed at either high or ambient pressures, and at high temperatures, in any of the project fluids A, B, F, G and I. However, physicochemical effects are identified, although none of these has demonstrated deterioration at a catastrophic rate; any property losses occur over a reasonable period of time at elevated temperatures. Unlike Coflon, Tefzel is more likely to exhibit reduced properties in aromatic oil than in methanol.

All of the ageing effects have resulted in gas permeation rate increases, especially with Fluid I.

## **1     INTRODUCTION**

In a similar approach to that used for the previously issued correlation report for Coflon (CAPP/M.10), this report aims to identify any correlations between mechanical property changes and chemical/morphological changes for Tefzel, using information supplied in other MERL and TRI project reports (plus latest data which will be included in final reports for Phase 1). Differences identified with Coflon behaviour will be of scientific interest as well as appropriate to project applications, as Tefzel and Coflon are chemical isomers.

Owing to the considerable chemical resistance of Tefzel, much of its testing so far has been based on mechanical properties. Where changes have occurred, chemical analysis can now be targeted more effectively.

Relevant test data collated here include: tensile modulus and related properties, permeation coefficients, % crystallinity, and other observations where significant.

Fluids based on methanol and amine (Fluid G), a mixture of methane, carbon dioxide and hydrogen sulphide gases plus an aqueous amine solution (Fluid F), and an aromatic oil mix of heptane, cyclohexane, toluene and 1-propanol (Fluid I) have affected Tefzel to varying degrees, and are discussed in some detail herein.

## **2     COMMENT**

The major use of Tefzel (ETFE) in offshore oil production is as umbilicals. Hence its chemical resistance to methanol is of particular importance. Other umbilical materials are: Hytrel, which has suffered stress crazing under some conditions, and Nylon 11. One possible reason why Tefzel has not been employed for flexible pipes thus far is its tendency (unlike Coflon) to fracture internally ('craze') during explosive decompression testing. Although such tests have not been a direct part of the CAPP project, this observation has been made when removing testpieces generally from the various high pressure gas exposures.

The inherent chemical inertness of ETFE is such that, to date, a suitable solvent has not been found to make the solutions necessary for GPC measurements. Hence assessment of any chemical changes which alter molecular weights has been impossible. X-ray diffraction studies are beginning to reveal changes in crystallinity due to ageing. However, most of the data generated so far have been from mechanical properties. This report will be updated if data from more chemical analyses become available.

### 3 RESULTS AND DISCUSSION

Unlike Coflon, Tefzel can be easily processed without the need for plasticizer. This should make the definition of baseline unaged Tefzel properties a great deal easier and, in the main, does so. However, TGA work has shown that the first 2% of Tefzel weight loss during this technique is associated with a lower activation energy than the remainder (although the value was still high); 2% of low molecular weight material was thus inferred. As will be seen, this low molecular weight species (modifier - perhaps termonomer) is apparently leached out during ageing in an analogous way to plasticizer loss for Coflon. Consequent effects on properties are addressed in this review.

Effects of the various fluids employed are now reviewed in turn for Tefzel. Those fluids which might be expected to react chemically with Tefzel are considered before those where only physical or physicochemical effects might be anticipated. In general, the comments concerning comparisons across tests made in section 2 of CAPP/M.10 also apply for comparisons made herein. More specifically, for expediency most of these have involved tensile test data, although more information might be expected from dynamic fatigue tests: however, the time required for the latter has restricted the number of tests conducted. Where they have been performed, crack growth resistance (CAPP/M.10) has been used to quantify each set of results in a single value for comparison purposes.

Tensile test values are useful for assessments of property changes (e.g. for modulus) brought on by exposure to a fluid, but they should not be compared directly with Coflon values elsewhere because Tefzel testpieces were only half the thickness of Coflon specimens, and thickness is known to affect tensile behaviour.

#### 3.1 Fluid F (wet H<sub>2</sub>S mixture + amine)

Fluid F is: 94/5/1 CH<sub>4</sub>/CO<sub>2</sub>/H<sub>2</sub>S gases saturated with water vapour, the water originally-added containing 1% of ethylene diamine. All exposures were carried out in the vapour phase of the mixture.

Table 1 summarises the property and analytical results for Fluid F 5000psi exposures of Tefzel at a variety of temperatures and test durations, and Figures 1 to 3 plot some of the tensile data from this table.

##### 100C exposures

Fluid F ageing at 100C initially caused a slight decrease in Tefzel's modulus after 1 month followed by a slight increase after 3 months (but not back to the original value, see Figure 1). A reduction in crystallinity (to 18% from 26%) had occurred after 1 month at 100C. HP methane permeation rate after 3 months had doubled from the unaged rate, and increased even more after 6 months. This has in the past been attributed to loss of low molecular weight components making permeation easier, but it is probably also largely influenced by the increase in amorphous levels (arising from the crystallinity loss) to assist diffusion of permeating fluids. More crystallinity measurements would be required to resolve this point.

### 120C exposures

The beginnings of a pattern for the change in modulus with exposure time is apparent after 120C exposures (see Figure 2). Once again, a minimum value is reached before the modulus increases again towards its original unaged value, although there is some variation near the minimum.

Crystallinity levels were again reduced (to 22.9%) after 4 weeks at 120°C compared to 26% for the unaged material.

Methane permeation at 150C and 5000 psi had again doubled after 4 weeks at 120C.

### 140C exposures

Clear evidence of a minimum modulus value is shown in Figure 3. After about 1 week's exposure to Fluid F at 140C, the modulus decreased by nearly a quarter from its original value. During the subsequent 3 week exposure, the modulus increased again, almost attaining its original value again.

Crystallinity level after 8 days exposure was 20.2%, a drop of 5.8% from the unaged value.

HP methane rate had again more than doubled, even after only 8 days' exposure.

### General

As modulus dropped to a minimum value at each exposure temperature, plotting log reciprocal time to this minimum against reciprocal absolute temperature should give a straight line relationship. The plot obtained (Figure 4) was approximately linear, with an activation energy of 8kcal/mol. It is likely that the 1/t 120C value should be greater (cf variation around minimum in Figure 2), so this activation energy is probably representative. This value indicates strongly that the change is probably not of a chemical nature.

The initial changes in modulus may be related to losses in crystallinity. The subsequent increase in modulus is sensibly associated with the slow loss of the 2% of low molecular weight species present. Insufficient X-ray diffraction data are available at present to substantiate this picture more fully by obtaining similar Arrhenius plots from the diffraction results. More values at longer times for each of the temperatures (and at a shorter time for 120C) are necessary.

The two crack resistance measurements - obtained for unaged and well-aged (4 weeks 140C) testpieces - were very similar (as were the mechanical properties for these testpieces).

The observations above, plus the similar elemental analysis values for % fluorine and the lack of change in appearance, indicate that little chemical change has occurred to Tefzel in any of the Fluid F exposures employed. However, physical or physicochemical changes (i.e. removal of low molecular weight species, loss of crystallinity) have taken place, although in a 'measured' fashion, i.e. no catastrophic deterioration has occurred.

### 3.2 Fluid G (methanol + 1% ethylene diamine)

As this fluid is 100% liquid, pressurisation was carried out with a liquid pump and all the testpieces were fully submerged.

Table 2 summarises Fluid G results obtained. Exposure at 120C gave a reduction in modulus with time, as shown also in Figure 5. No modulus increase at longer times (cf Fluid F results), and hence no minimum modulus values, have been observed to date. Yield strain had increased from the original value of 20%, to 30% after 6 weeks. This ductile failure was in complete contrast to Coflon under these conditions, which failed in a brittle manner (CAPP/M.10). Modulus etc behaviour at the other temperatures supported this trend.

Crystallinity of the 4 week aged samples showed a massive drop to 1.7% from 26%. The one-off nature of this measured value means that it needs verifying with other similarly-aged samples.

HP methane permeation has increased after ageing in Fluid G, as it did for Tefzel after ageing in any of the fluids. Once again, permeation rate had doubled. This tends to indicate a common effect as discussed for Fluid F.

No colour changes or cracking of Tefzel were observed such as those observed for Coflon in Fluid G. Hence again the inferences are that physicochemical effects, but not actual chemical changes, are associated with Fluid G exposures. However, it may be less controllable than for Fluid F, as steady decreases in mechanical properties have been observed.

### 3.3 Fluid I (high aromatic oil)

The constituents of Fluid I and their relative proportions are: heptane/cyclohexane/toluene/1-propanol, 35/35/20/10 by volume.

Again testpieces were fully submerged in the liquid: all tests were conducted at 140C. However, as well as testing at 5000 psi 140C, one test cell was run at vapour pressure, *i.e.* a gap was left at the top of the cell which would be saturated with vapour at 140C: no extra hydraulic pressure was applied in this case.

A large reduction in modulus occurred after 2 weeks at 140C 5000 psi (Table 3), the samples having lost half of their original modulus. An increase in modulus occurred after the 10 week exposure, but this had decreased somewhat again after 30 weeks. At vapour pressure no measurement was taken at 2 weeks, but at 10 weeks an even greater loss in modulus was recorded compared to the 5000 psi case, and at 30 weeks the value was even lower. Some swelling of the sample was also observed in this vapour pressure case only; clearly the constraining hydraulic effect at 5000psi opposed the swelling somewhat.

At 10 weeks, the application of 8% strain made little difference to measured values.

The value of crack resistance after 30 weeks at 140C was greater than the unaged value, in contrast to the modulus behaviour. Strain again made little difference.

Crystallinity again showed a large decrease falling from 26% to 9% after 10 weeks at 140C. Once again, no colour change was noted.

HP permeation rate was increased even more by Fluid I exposures than by Fluids F and G, the effect increasing with exposure time. The maximum increase was over four-fold the unaged value.

### **3.4 Fluids B (wet CH<sub>4</sub>/CO<sub>2</sub>) and A (methanol)**

Because even those fluids considered to date (which are more chemically-aggressive than Fluids B and A) did not cause chemical deterioration of Tefzel, little change would be expected here. This conclusion is confirmed by the unchanging elemental analysis values and unaltered appearance of Tefzel in the fluids B and A.

In both cases, modulus decreases with time were observed (presumably again due to crystallinity loss although appropriate values are not available). Comparison of the 2 week and 4 week module at 140C perhaps suggests that the loss of low molecular weight species is beginning to show for Fluid B. HP methane permeation results are again doubled.

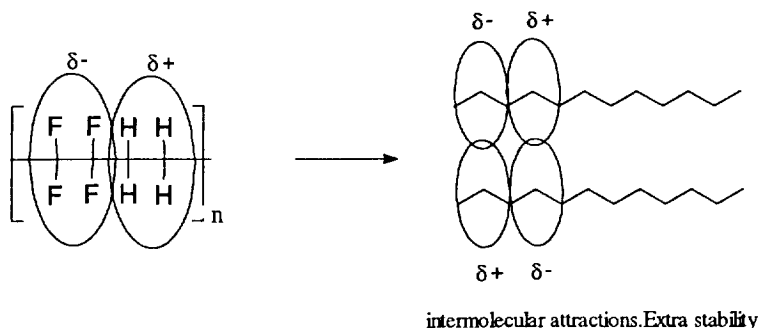


#### 4 FURTHER DISCUSSION

To commence assessing the overall picture for Tefzel fluid exposures, although, as discussed below, solubility parameters  $\delta$  are not major factors for this material - no common solvent dissolves Tefzel - nevertheless Fluid I with a  $\delta$  value of  $8.5 \text{ (cal/cm}^3)^{1/2}$  is seen to be more aggressive in swelling terms than methanol ( $\delta$  14.5), in contrast to the Coflon ( $\delta$  11.3) situation. Presumably the  $-\text{CH}_2\text{CH}_2-$  groupings reduce  $\delta$  compared with the Coflon value. Another interesting parallel for Tefzel (a TFE/ethylene copolymer) is the behaviour of the elastomer Aflas, a TFE/propylene copolymer, which swells up to 30% in hydrocarbons. The overall performance of Tefzel in the project fluids is apparently governed not by chemical reaction, but by resistance to physicochemical attack. Because the worst attack of this type is by a main oilfield production fluid, another reason (besides explosive decompression) is now given for its lack of use in flexible pipes. additionally, its successful employment with methanol in umbilicals can be seen as reasonable: umbilicals would not be used at 140C, where some Tefzel modulus decrease was seen with methanol (Fluid A), and even at this temperature there is no sign of the catastrophic deterioration which can be suffered by Coflon in methanol.

Turning to the mechanical and chemical stability of Tefzel, this can be attributed to several factors<sup>1</sup>. If we compare ETFE (Tefzel) with PTFE (Teflon) which is fully fluorinated, some of the reasons for the stability become apparent. Substitution of some fluorine in PTFE by hydrogen in a regular alternating manner (pairs of atoms) to give ETFE results in polarity, which leads to enhancement of mechanical properties. Polarity occurs because hydrogen has a different electronegativity compared to fluorine, and a different bond length to carbon. The centres of electronegativity and positivity *along* the polymer chain become unbalanced but can become more balanced *between* chains, given the correct alignment, see (1) below. No covalent bonds are broken in forming stabilised ETFE. Increased interaction between polymer chains results in increased mechanical properties compared to PTFE.

(1) Electron density distribution for ETFE

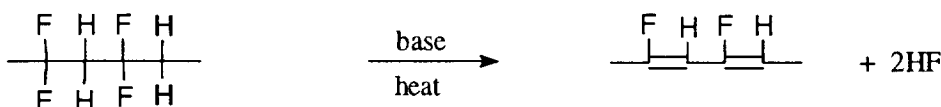


Polarity and interpolymer chain attraction also influence fluid penetration into the amorphous regions of the polymer, explaining why Tefzel has no known solvent at ordinary conditions. Even with chemically-similar solvents (e.g. the solvents and Tefzel have similar  $\delta$  values), the implied thermodynamic consequence of these interchain attractions is that

entropy changes are kept to such an insignificant level that they overrule enthalpic effects (associated with  $\delta$ ) which normally produce a negative free energy, and hence fluid absorption. Similar effects for polymers generally can be obtained for other structural reasons, for instance chain crosslinking.

These attributes contrast well with the chemical behaviour of PVDF (Coflon) reported in CAPP/M.10. Several chemical mechanisms for Coflon deterioration are outlined in that report. The abundance of adjacent H and F atoms in PVDF leads to dehydrofluorination and formation of a chemically stable conjugated molecule, see (2) below.

## (2) Conjugated PVDF



The hypothesis<sup>1</sup> for the chemical stability of Tefzel, and the potential for possibly being attacked of Coflon, are again supported by a structural argument. The stabilized structure for Tefzel in (1) means that double  $-\text{CF}_2\text{CF}_2-$  groups align with analogous  $-\text{CH}_2\text{CH}_2-$  groups on neighbouring chains - not the best arrangement for the occurrence of crystallization, which has been found to be at a level of only 26%, even when unaged. For Coflon, single  $-\text{CF}_2-$  groups and  $-\text{CH}_2-$  groups would align for interactive stability between chains: geometrically, however, only a small adjustment is required for each  $-\text{CF}_2-$  group to align with another  $\text{CF}_2$  group on an adjacent chain, so that crystallization can occur, but at the expense of interchain chemical stabilization. In this project, crystallinity levels of ~50% have been measured for Coflon. Hence the greater drive for crystallinity apparently reduces interchain chemical stability for Coflon, whilst the reverse is true for Tefzel. These basic tendencies influence the whole fabric of mechanical property and fluid absorption for the two polymers.

Increased HP methane permeation through aged Tefzel has been attributed to (a) an increased volume of amorphous region present (i.e crystallinity loss), and (b) removal of 2% low molecular weight species. The second concept infers that "holes" left as low MW polymer is removed are maintained, to increase permeation; for this, some interaction between chains to maintain the microporous structure must exist. The interchain attractions discussed above could perform such a function.

## 5 **GENERAL CONCLUSIONS**

- No evidence has so far been found from mechanical property, crack growth resistance and chemical analysis tests for any chemical degradation of Tefzel during exposures to the project fluids at severe conditions.
- Intermolecular attractions between adjacent polymer chains are believed to account for the chemical stability of Tefzel as well as resulting in good mechanical behaviour. Reasons why these do not apply to Coflon, and possibly-related crystallinity aspects, have been discussed.
- Evidence has been found for physicochemical changes: reductions in crystallinity levels have been measured, and a loss of 2% of low molecular weight polymer has been implied by the data. Further analysis needs to be conducted to verify whether loss in crystallinity can be correlated with any physical properties, but initial observations indicate that this is quite likely.
- Modulus has been shown to fall to a minimum and then rise slightly after exposure to Fluid F. A relationship was observed between this feature and the temperature of exposure.
- Fluid G ageing has resulted in a gradual decline in modulus values during 120C exposures.
- A large decrease in modulus has been observed during Fluid I exposures at 140C.
- Increased HP methane permeation through aged Tefzel has been attributed to increases in amorphous content and loss of low molecular weight species leaving a microporous structure. Fluid I caused a four-fold permeation increase, the other fluids double the unaged rate: the rate also increases with exposure time and temperature.
- It is likely that the solubility parameter of Tefzel is lower than that of Coflon, so that aromatic oil (Fluid I) can be more aggressive than methanol to Tefzel, but the reverse is true for Coflon.
- Modulus measurements may reflect crack growth resistance for HP gas exposures, but not liquid exposures.

## **6 ONGOING AND FUTURE WORK**

For the remainder of Phase 1, a limited number of selected tests for Tefzel will be performed for completion in some areas. A few other tests are ongoing in anticipation of Phase 2, in which long-term tests are an essential target. The list is:

- 1 Work involving Fluid F  
Long term 85C 5000 psi exposures: 2 months to 2 years.  
Modulus, analysis, etc.
- 2 Work involving Fluid G  
Exposures at 65C, and 85C.  
Modulus, analysis, etc.

In the event of Phase 2, a new and full programme will be drawn up. This could include a continued investigation into finding a solvent for Tefzel for GPC analysis.

## **ACKNOWLEDGEMENTS**

This report is a resume of data and ideas from all members of the CAPP project team at MERL and TRI. Sources of information are the CAPP technical reports, progress reports, meeting notes and personal discussions.

## **REFERENCE**

- 1 J F Imbalzano, Basic Chemistry and Properties of Teflon/Tefzel, Technical Information Bulletin, Du Pont (USA)

**TABLE 1 Tefzel aged in Fluid F(CH<sub>4</sub>/CO<sub>2</sub>/H<sub>2</sub>S + 1% ethylene diamine in H<sub>2</sub>O) at 5000psi, unstrained**

Sample & ageing history	Modulus	Yield stress	Yield strain	Ult strength	Ult strain	70C crack growth resistance (J for 10nm/c)	CH <sub>4</sub> perm'n coeff Q	Crystallinity	Elemental analysis	Visual appearance
	(MPa)	(MPa)	(%)	(MPa)	(%)	(kJ/m <sup>2</sup> )	(cm <sup>2</sup> /s/atm) x 10 <sup>-6</sup>	(%)	(%F)	
Unaged	910	24	20	42	427	3.3	0.07	26.0	61	Translucent, white
2 month 85C	927	27	18	42	402		-	-		
1 month 100C	863	26	20	>31	>245		-	18.0	60	)
3 months 100C	897	25	20	>30	>240		0.14	-		)
6 months 100C	-	-	-	-	-		0.18	-		)
1 week 120C	798	23	20	42	434		-	-		)
2 weeks 120C	841	25	22	>29	>250		0.12	-		) Virtually
3 weeks 120C	775	22	20	39	414		-	-		) unchanged
4 weeks 120C	811	26	20	39	369		0.15	22.9	61	)
6 weeks 120C	865	24	21	35	358		TBT	-		)
3 days 140C	790	23	23	>30	244		-	-		)
5 days 140C	737	23	25	>30	>250		-	-		)
8 days 140C	714	23	24	>27	>250		0.15	20.2	61	)
2 weeks 140C	892	23	20	39	390		-	-		)
4 weeks 140C	900	24	21	38	382	3.4	0.18	-		)

NB: Modulus etc from tensile measurements  
 CH<sub>4</sub> permeation at 150°C, 5kpsi  
 TBT, to be tested before the end of Phase 1



**TABLE 2 Tefzel aged in Fluid G (methanol + 1% ethylene diamine), unstrained at 5000psi unless indicated otherwise**

Sample & ageing history	Modulus (MPa)	Yield stress (MPa)	Yield strain (%)	Ult strength (MPa)	Ult strain (%)	CH <sub>4</sub> perm'n coeff Q (cm <sup>2</sup> /s/atm) x 10 <sup>-6</sup>	Crystallinity (%)	Visual appearance
Unaged	910	24	20	42	427	0.07	26.0	White
8 weeks 65C at vapour pressure	897	26	20	45	458			)
8 weeks 85C	873	27	21	41	410			)
10 weeks 85C	828	26	22	40	385			) Slight
12 weeks 85C	773	26	25	41	398			) discoloration
6 weeks 100C	803	26	25	41	391			)
8 weeks 100C	797	26	23	39	414			)
2 weeks 120C	804	23	21	39	399			)
4 weeks 120C	805	25	24	41	448	0.15	1.7?	)
6 weeks 120C	755	24	30	41	466	0.18		)

NB: Modulus etc from tensile measurements  
CH<sub>4</sub> permeation at 150°C, 5kpsi

**TABLE 3 Tefzel aged in Fluid I (heptane/cyclohexane/toluene/1-propanol), unstressed at 5000psi unless indicated otherwise**

Sample & ageing history	Modulus	Yield stress	Yield strain	Ult strength	Ult strain	70C crack growth resistance (J for 10nm/c)	CH <sub>4</sub> perm'n coeff Q	Crystallinity	Visual appearance
Unaged	(MPa)	(MPa)	(%)	(MPa)	(%)	(kJ/m <sup>2</sup> )	(cm <sup>2</sup> /s/atm) x 10 <sup>-6</sup>	(%)	
2 weeks 140C	910	24	20	42	427	3.3	0.07	26.0	White
10 weeks 140C	444	19	54	28	256		-		)
10 weeks 140C	551	19	25	30	333		0.23	9	)
8% tensile strain	583	19	21	36	433				)
10 weeks 140C at vap pressure	517	18	26	29	351				) Unchanged
30 weeks 140C	518	20	42	30	341	4.2*	0.30	-	)
30 weeks 140C at vapour pressure	494	20	35	30	350				)

NB: Modulus etc from tensile measurements

CH<sub>4</sub> permeation at 150°C, 5kpsi

\* When tested strained at the same conditions, crack growth resistance = 4.0

**TABLE 4 Tefzel aged in Fluid B (methane, carbon dioxide, with saturated water vapour) at 5000psi**

Sample & ageing history	Modulus (MPa)	Yield stress (MPa)	Yield strain (%)	Ult strength (MPa)	Ult strain (%)	CH <sub>4</sub> perm'n coeff Q (cm <sup>2</sup> /s/atm) x 10 <sup>-6</sup>	Crystallinity (%)	Elemental analysis (%F)	Visual appearance etc
Unaged	910	24	20	42	427	0.07	26.0	61	Translucent, white
12 weeks 90C	772	24	21	34	357				)
4 weeks 120C	800	23	21	38	400				)
12 weeks 120C	745	24	21	38	400				) No change
2 weeks 140C	798	23	21	36	379	0.16		61	)
4 weeks 140C	800	22	21	36	388				)
									)
									)

NB: Modulus etc from tensile measurements  
CH<sub>4</sub> permeation at 150°C, 5kpsi



**TABLE 5 Tefzel aged in Fluid A (methanol), unstrained at 5000psi unless otherwise indicated**

Sample & ageing history	Modulus (MPa)	Yield stress (MPa)	Yield strain (%)	Ult strength (MPa)	Ult strain (%)	CH <sub>4</sub> perm'n coeff Q (cm <sup>2</sup> /s/atm) x 10 <sup>-6</sup>	Crystallinity (%)	Elemental analysis (%F)	Visual appearance etc
Unaged	910	24	20	42	427	0.07	26.0	61	Translucent, white
6 days 140C at vapour pressure	-	-	-	-	-	-	25.6		
8 days 140C	862	26	22	38	350			61	)
14 days 140C	715	24	20	35	351				) Unchanged
14 days 140C 3% tensile strain	700	24	20	38	384				)
14 days 140C 7.5% tensile strain	670	26	22	36	327	0.15*			)

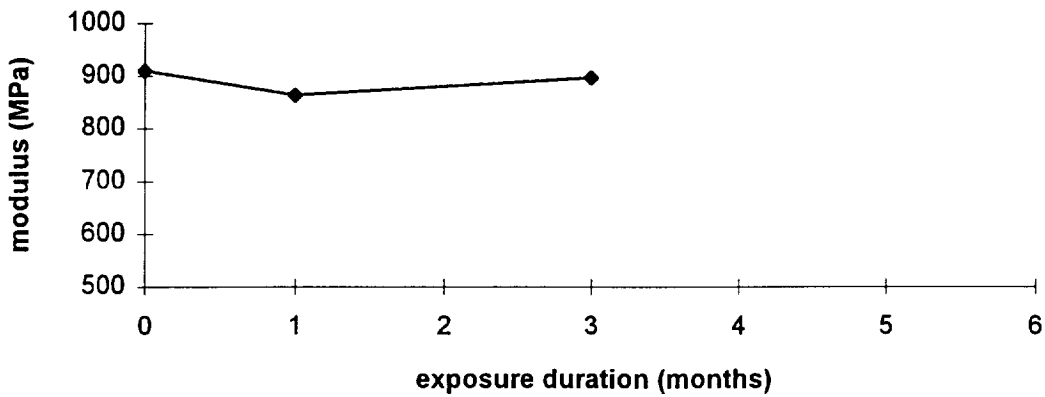
NB: Modulus etc from tensile measurements

CH<sub>4</sub> permeation at 150°C, 5kpsi

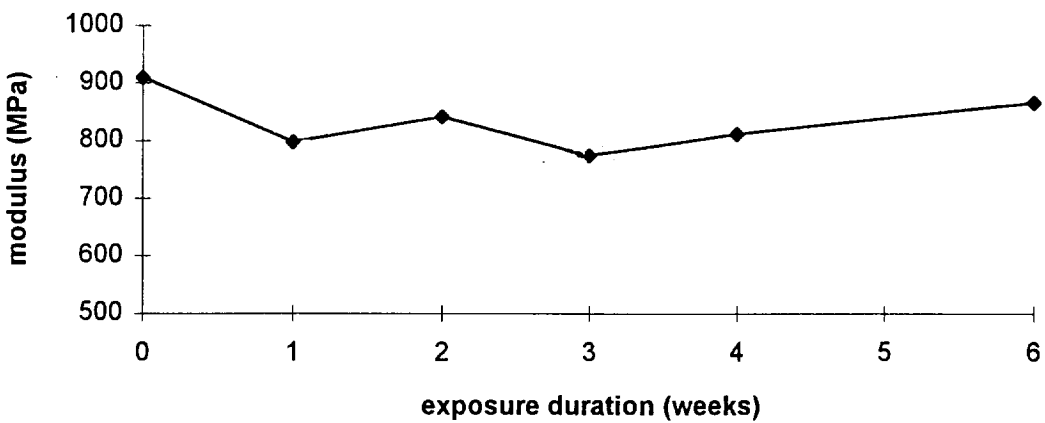
The diffusion coefficient at 150C for Fluid A ingress into unaged Tefzel is 0.25x10<sup>-6</sup>cm<sup>2</sup>/s

\* Aged at 150C

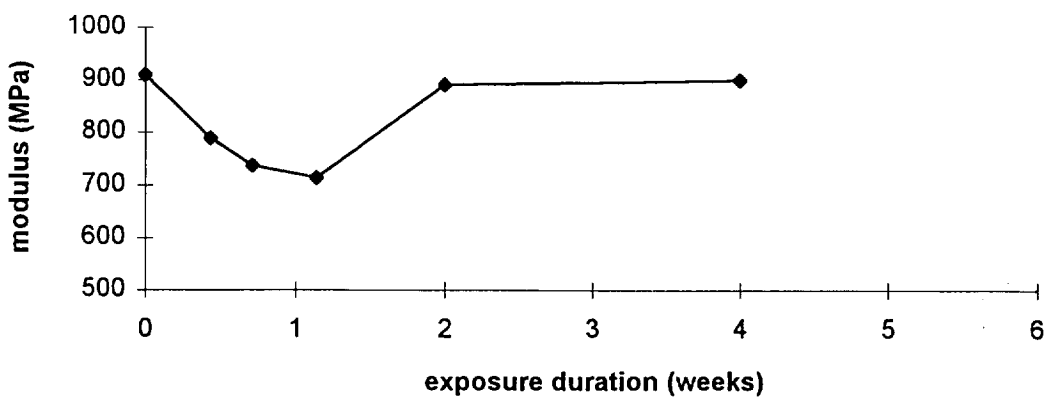
**FIGURE 1: Change in modulus of Tefzel exposed to Fluid F at 100'C**



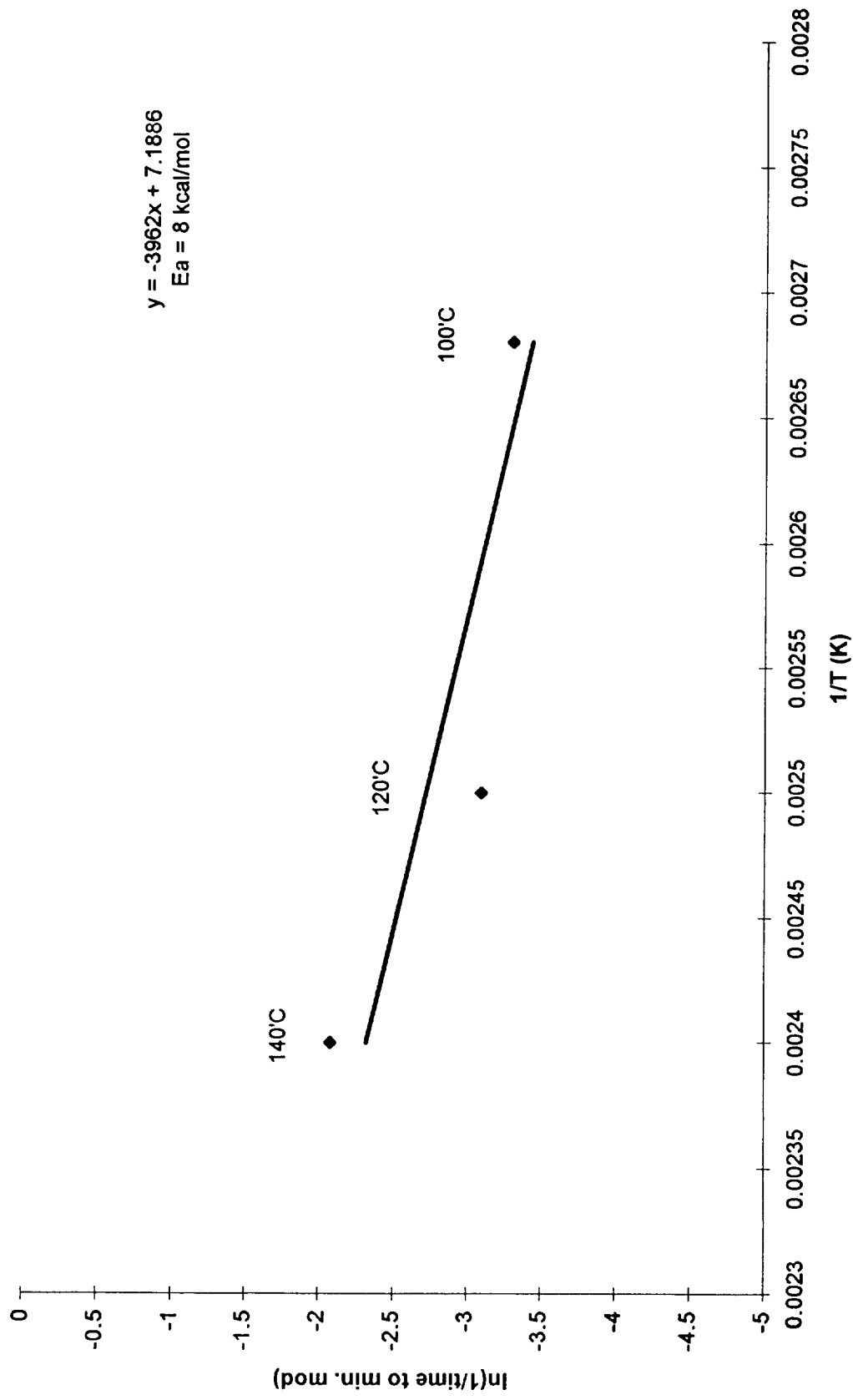
**FIGURE 2: Change in modulus of Tefzel exposed to Fluid F at 120'C**



**FIGURE 3: Change in modulus of Tefzel exposed to Fluid F at 140'C**



**FIGURE 4: Arrhenius plot of time to minimum modulus  
after Fluid F exposure of Tefzel**



**FIGURE 5: Change in modulus of Tefzel exposed to Fluid G at 120°C**

